



Publication 463-013

1988

A REVIEW OF  
POTASSIUM BITARTRATE STABILIZATION OF WINES

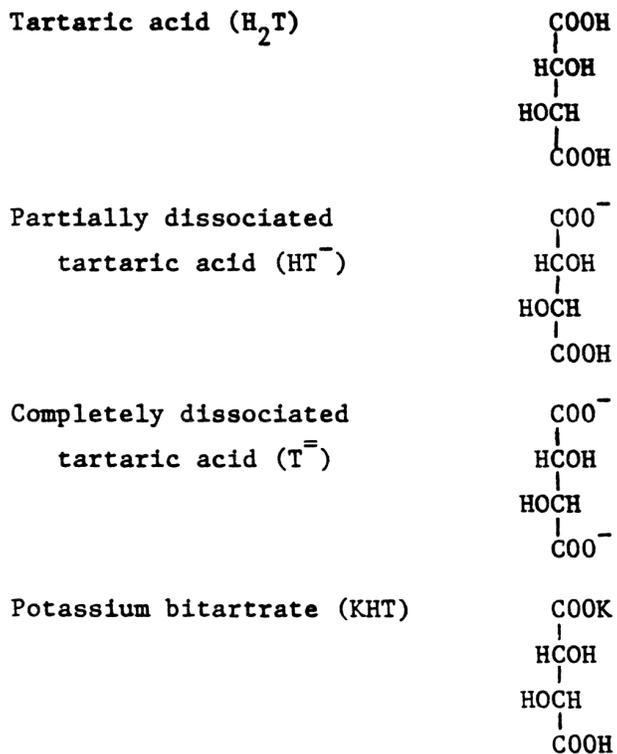
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Wine stability considerations can be classified as: microbiological, protein, tartrate, red wine oxidative and color, white wine oxidative and color, and metals. The problems of producing stable, highly palatable products are of concern to every vintner. The days when wineries can release wines to the public with physical instabilities and still compete has long past. It is essential that every vintner understand the parameters affecting wine stabilities and operate to obtain product stability while maximizing palatability.

Potassium Bitartrate Instability. Tartaric acid (H<sub>2</sub>T) and its salt, potassium bitartrate (KHT), are considered normal constituents of wine. The various ionization forms of tartaric acid found in grapes and wines are:



Potassium bitartrate (KHT) is believed to be produced after veraison with the movement of potassium from the soil into the fruit. During ripening, the amount of undissociated tartaric acid decreases as the mono and dibasic potassium salts are formed. Simultaneously, tartaric acid is diluted at a rate depending on the variety and climatic conditions. Additionally, the tartrate content is also influenced by location, cultural practices, and the state of maturity.

The potassium uptake of the vine is dependent on factors such as rootstock, soil type and depth, irrigation treatment, etc. Thus, both tartrate and potassium contents differ widely with variety, region, climate, and viticultural practices. As a consequence, the enologist handles juices and wines that have large variations in KHT concentrations.

All wines differ in their "holding" or retention, capacity for tartrate salts in solution. If the holding capacity is exceeded, these salts will precipitate, resulting in the formation of "tartrate casse". Solubility of potassium bitartrate is dependent primarily upon the alcohol content, pH, the temperature of the wine, and the interactive effects of various cations and anions.

Although KHT is soluble in grape juice, the production of alcohol during fermentation lowers the solubility and gives rise to a supersaturated solution of KHT. In wine, the following equilibrium exists:

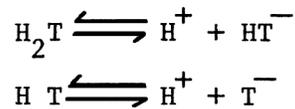
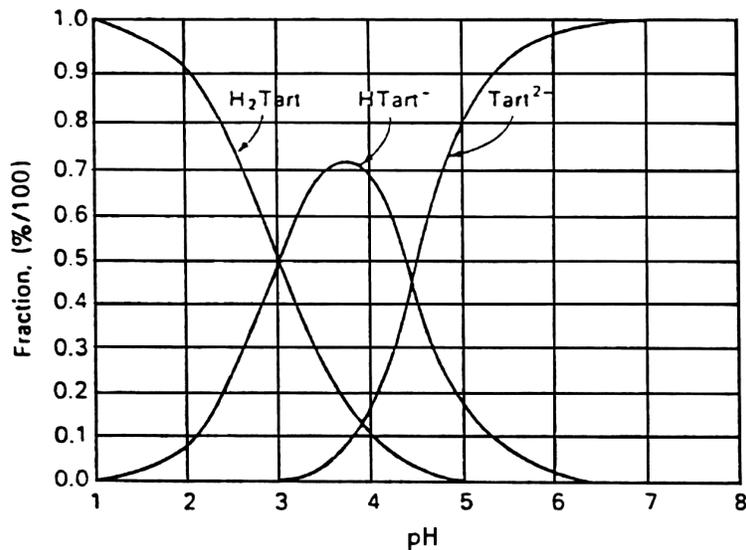


Figure 1 shows the distribution of tartrate species in wine as a function of pH. The percentage of tartrate present as potassium bitartrate (KHT) is maximum at pH 3.7, and, if other factors permit, precipitation will be greatest at this point. Thus, any wine treatment causing changes in pH, such as blending, occurrence of a malolactic fermentation, etc., may affect subsequent bitartrate precipitation. This is a primary reason why potassium bitartrate stability must be checked following all cellaring activities, just prior to bottling.

Figure 1. Tartaric acid species as a function of pH (Fritz and Schenk 1974)



Tartrate stability has been traditionally accomplished by chilling, ion exchange, or both. A number of new methods to obtain potassium bitartrate stability have been attempted. These include filtration (Scott et. al 1981), electrodialysis (Postel et. al 1977), reverse osmosis (Wucherpfennig 1978), crystal flow (T.M.) (Riese 1980), and contact seeding. Of these processes, filtration, crystal flow (T.M.), and contact have been the most successful commercially (Ewart 1984). In conventional cold stabilization procedures (chill-proofing), wines are chilled to a temperature designed to decrease potassium bitartrate (KHT) solubility, which optimally results in precipitation. The most important variables affecting the precipitation of potassium bitartrate during chilling are: 1) the concentration of the reactants, specifically tartaric acid, 2) the availability of foci or nuclei for crystal growth, and 3) the solubility of the potassium bitartrate (KHT) formed. Perin (1977) determined the following relationship for determination of the temperature needed for KHT precipitation:

$$\text{Temperature } (-^{\circ}\text{C}) = \left( \frac{\% \text{ alcohol}}{2} \right) - 1$$

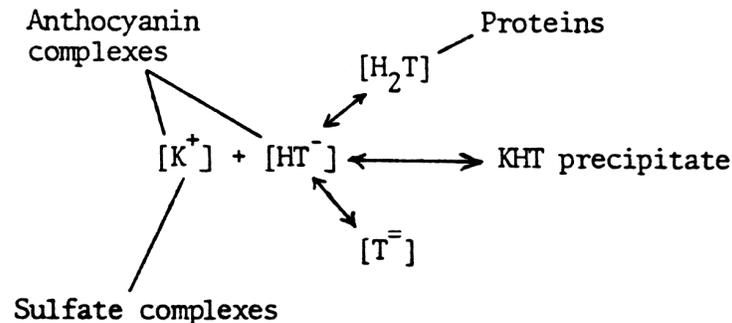
KHT precipitation occurs in two phases. The induction phase is when the level of KHT in solution increases due to chilling. This is followed by the crystallization phase where crystal growth and development occurs. The rate of precipitation of potassium bitartrate at low temperatures is more rapid in table than in dessert wines and more rapid in white than red wines (Marsh and Guymon 1959). During conventional chill-proofing, precipitation is usually rapid during the first 12 days, then the rate of KHT precipitation diminishes considerably. This reduction is due to a decreased level of KHT saturation in solution. Temperature fluctuations during cold stabilization can have a significant effect in reducing the rate of KHT precipitation due to the large effect this has on the speed of nuclei formation. Without crystal nuclei formation, crystal growth and subsequent precipitation cannot occur. Simply opening the cellar doors in the winter, although cost effective, may not provide optimal temperatures for KHT precipitation or product palatability of certain wines. The increased absorption of oxygen into the wine at low temperatures over longer periods, and the subsequent oxidation of wine components, makes alternatives to conventional cold stabilization desirable.

Koldone is a proprietary product (Cellulo Corporation) used to help obtain potassium bitartrate stability. It is produced by mixing calcium carbonate and L(+) tartaric acid in a specific ratio. The addition of Koldone into a wine causes the added calcium carbonate to react with the wines tartaric acid to form an insoluble calcium tartrate. Precipitation is aided by calcium tartrate crystals present in Koldone, which act to seed. Thus, the addition of Koldone to a wine causes it to become super-saturated with respect to calcium tartrate and this is precipitated rapidly from solution. The tartaric acid concentration can be reduced to a low enough level where cold stability is achieved. For a more detailed discussion see Clark et al. (1985). Several other proprietary compounds designed to aid potassium bitartrate stability are available.

"Complexing factors" can greatly affect potassium bitartrate formation and precipitation, see figure 2. Wine is able to support a supersaturated solution of KHT because a percentage of the tartrate and potassium ions is thought to be complexed with wine constituents and therefore not precipitated. Metals, sulfates, proteins, gums, polyphenols, etc., can form complexes with free tartaric acid and potassium ions, thus inhibiting the formation of KHT (Pilone, et. al. 1965). These complexes formed are mainly between polyphenols and tartaric acid in red wines and proteins and tartaric acid in whites. The binding of free tartaric acid

prevents potassium bitartrate formation. In a study of white Bordeaux wines, Peynaud *et al.* (1964) found sulfate to be the most important factor in stability besides free potassium or tartrate. This would appear to be due to the complexing ability of sulfate with potassium (Chlebek and Lister 1966). Almost one half of the sulfate in white wines and 100% of the sulfate in red wines is hypothesized to complex free potassium as  $K_2SO_4$  or  $KSO_4^-$  (Bertrand *et al.* 1978). The differences in the extent of tartrate complexes formed and their "holding power" to prevent precipitation depends upon the wine in question.

**Figure 2**  
**Potassium bitartrate equilibria and the interaction of "complexing factors".**



Pigments of red wines are often involved in complexes with tartaric acid (Balakian and Berg 1968). As wine oxidizes and pigment polymerization occurs, the holding capacity for tartaric acid diminishes, often resulting in delayed precipitation of potassium bitartrate. Additionally, cultivar and growing region may affect potassium bitartrate holding capacity (Boulton 1980). Each wine, because of its unique composition, will achieve and equilibrium of KHT solubility under imposed temperature conditions.

Pilone and Berg (1965) and Balakian and Berg (1968) have suggested the importance of colloids as affecting potassium bitartrate stability. It is postulated that pectins and other polysaccharides, such as glucane from *Botrytis cinerea*, inhibit crystallization. This may be the result of these compounds absorbing onto the surface of the crystal and preventing further crystal growth. Neradt (1977), however, in a study of German white wines could find no inhibition of crystallization by either gelatin or gum arabic. Further research in the area would be welcomed.

Occasionally, winemakers choose to add complexing factors or inhibitors to prevent potassium bitartrate formation. In theory, the right inhibitor in the proper concentrations could reduce the need for traditional chill proofing, bitartrate seeding, or ion exchange by preventing the formation of potassium bitartrate crystals. Unfortunately, none of the approved wine additives for such purposes are completely satisfactory.

Perhaps the best known inhibitor of potassium bitartrate crystal formation is metatartaric acid, the hemipolyactide of tartaric acid. This is an approved wine additive in certain countries, although not in the U.S. Metatartaric acid is produced by heating tartaric acid at  $170^\circ\text{C}$  for about 120 hours. It has been reported that 50 to 100 mg/l protects young wines from potassium bitartrate precipitation,

even when they are stored at low temperatures for several months (Peynaud and Guimberteau 1961). Cold water should be used for dissolving the metatartaric acid, to avoid its hydrolysis (Peynaud 1984). The mechanism of inhibition seems to be an interference in the formation and growth of potassium bitartrate crystals. This occurs due to the coating by metatartaric acid of the crystalline tartrate nuclei, which prevents their growth (Peynaud 1984). After adding to wine, metatartaric acid is slowly hydrolyzed to tartaric acid, with a corresponding loss of activity. Its period of effectiveness is a function of the storage temperature of the treated wine. Peynaud (1984) found wines stored at 0°C were inhibited from KHT precipitation for several years, but that metatartaric disappeared after two months in wines stored at 25°C. Therefore, metatartaric acid would have its greatest use in wines that are to be consumed rather quickly.

Carboxyl methyl cellulose is another inhibitor of potassium bitartrate precipitation (Cantarelli 1963). Additionally, purified apple pectin and tannin can inhibit crystal formation. One gram/l of tannin is said to strongly inhibit precipitation of tartrates (Wucherpennig and Ratzka 1967).

Rather than adding inhibitors, it is common to attempt to remove or reduce the complexing factors so that potassium bitartrate precipitation and stabilization can occur prior to bottling. Consequently, there is an intimate relationship between wine fining and potassium bitartrate stabilization. For example, it is known that condensed polyphenols interfere with tartrate precipitation (Amerine and Joslyn 1970). This suggests that removal of a portion of these polyphenols with certain fining agents prior to cold stabilization could enhance potassium bitartrate precipitation (see Zoeklein 1984, 1985).

Cold stabilization procedures (chill-proofing) cause precipitation of potassium bitartrate crystals as well as proteins. The proteins of white wines can exhibit a holding capacity for tartaric acid, thus inhibiting KHT formation (Piloni and Berg 1965). White wines contain relatively large insoluble proteins. As the phenols of white wines oxidize they polymerize and soon bind to and co-precipitate with proteins. This precipitation affects the tartrate holding capacity. White wines are usually too deficient in tannins to cause initial protein precipitation. Bentonite fining decreases the tartrate holding capacity of wines by removing both proteins and some wine phenols (Berg and Akiyoshi 1971). Additionally, if the wine pH is below 3.65, chill-proofing causes a downward shift in pH (Boulton 1980), also enhancing protein precipitation. This reduction in pH and the precipitation of proteins caused by cold stabilization is why some winemakers elect to fine with bentonite during or following potassium bitartrate stabilization. Bentonite fining during cold stabilization allows potassium bitartrate crystals to help compact bentonite lees. The addition of 2.5 to 10.0 pounds of bentonite per 1000 gallons reduces concentration product values of dry whites from 15 to 18%, and dry red CP values from 25-32% (Berg et. al 1968). Gorinstein et al. (1984) reviewed bentonite fining in conjunction with cold stabilization and the chemical changes that occur.

#### Determining Potassium Bitartrate Stability

There is significant variation in cellar activities employed to reach stability. Additionally, there is significant variation in the methods by which KHT stability is measured and defined.

Table 1 from Cooke and Berg (1984) shows the multitude of procedures used to determine cold stability by 18 California wineries surveyed. The fact that there is no industry standard for determination of cold stability is readily apparent

from their survey. This is not surprising in view of the fact that stability is a relative term that can and is defined differently by different producers.

Table 1

Winery	Test
A	Filter sample. .65 $\mu\text{m}$ membrane. Seed sample with KHT. 36 h at 25°F with mixing. analyze for tartrate
B	30 days at 30°F and CP.
C	14 days at 32-34°F. examine cold at 7 and 14 days.
D	Not done-wines stable due to prolonged aging in cool cellar.
E	Filter thru .45 $\mu\text{m}$ membrane and test 2 samples: A. 12 h frozen. 12 h at RT. examine daily. B. 72 h at 38-42°F. 12 h at RT. examine daily.
F	CP
G	96 h at 18°F. examine. 24 h at RT. examine. Deposits formed at 18°F should redissolve in 24 h at RT.
H	Filter sample thru .45 Nm membrane. A. 16 h at 10°F. thaw at RT. examine. B. 16 h at 15°F. thaw at RT. examine.
J	3-4 wk at 35°F. examine cold and RT.
K	Wine sample is pad filtered and membrane filtered if needed. A. One aliquot - 48 h at 26°F. a 2nd aliquot - 48 h at 100°F and both examined. B. Both aliquots of A then 48 h at 26°F and examined. and C. Both of B then 48 h at RT. and examined.
L	2 wk at 40°F. examine.
M	Filter. 24 h at 25°F. examine.
N	72-96 h at 40°F. examine or freeze 12 h. thaw. examine at RT.
P	Filter. 65 $\mu\text{m}$ membrane. freeze 24 h. thaw. examine at RT.
Q	4 h at 12°F. examine.
R	2 wk at 34°F. examine cold.
S	Not done.
T	12 h freeze and/or 72 h at 28°F. examine cold and at RT. Stability based upon amount of deposit.

Two traditional methods of determining potassium bitartrate stability are the determination of CP's (Concentration Product values) and the freeze or slush test.

The CP Test. The relationship between tartaric acid and potassium ions can be expressed quantitatively by using the Concentration Product (CP):

$$\text{CP} = (\text{moles/l K}^+) (\text{moles/l of total tartrates}) (\% \text{HT}^-)$$

Berg and Keefer (1958), using model water/alcohol solutions, were able to establish solubility levels for potassium bitartrate and, as a result, to calculate solubility produce values. Where calculated "CP values" exceed the published values considered "safe" for the wine type under consideration, the wine may deposit

tartrates. Conversely, if the calculated values are less than the identified safe levels, the wine is considered by some to be stable with respect to bitartrate precipitation. Therefore, determination of CP's may be a useful guide to the winemaker in estimating the probability of bitartrate instabilities. In Cooke's and Berg's review (table 1), only 2 of 18 California wineries surveyed used CP values for estimating potassium bitartrate stability. Any determination of CP values requires preliminary analysis of alcohol, pH, potassium ion, and total tartaric acid. CP tables are available in most wine texts, including Zoecklein et al. (1985).

The concentration product procedure does not allow for the complexing action of certain wine constituents that prevent crystal formation. This may be a particular problem for some wines.

Freeze Test. Another traditional method of measuring potassium bitartrate (KHT) stability is the freeze test. This procedure relies on the formation of crystals as the result of holding filtered wine samples at reduced temperatures for a specified time period. As indicated in Table 1 a wide variety of procedures are employed for this analysis. Often one of the samples is frozen and then thawed to determine the development of bitartrate crystals and if those crystals return to solution. The absence of crystal formation is meant to indicate a potassium bitartrate stable wine.

The freeze test is in reality a distorted crystallization-rate test (Boulton 1983). As ice formation increases, there is an increase in the relative concentration of all species in the sample, including alcohol, thus enhancing nucleation and crystallization. It is difficult to accurately relate crystal formation in this concentrated wine sample with bitartrate potassium instability. As such, the freeze test is essentially a crystal-rate test rather than an actual stability test. Filtration of the laboratory sample will remove crystal nuclei, which will affect test results. Unless one provides seed crystals (or doesn't lab-filter the test sample), precipitation over the short time period of the test is in fact a measure of the ability to form nuclei and precipitate. As Clark et al. (1985) report, two wine samples may be equally unstable. However, upon filtration and storage at low temperature, one precipitates tartrate and is considered unstable, whereas the second does not form nucleation sites at the same rate and is by way of this test considered stable. As stated, the presence of crystals depends upon the rate of crystallization and time. The relationship between the results of a freeze test and long-term bottle stability is therefore vague.

A more accurate determination of potassium bitartrate stability can be determined by seeding with potassium bitartrate. The oversaturated portion of tartaric acid and potassium is deposited onto the added crystals. There is a reduction in the tartaric acid, potassium, titratability acidity, and electrical conductivity. These changes can be measured to determine potassium bitartrate (KHT) stability.

Conductivity Testing. Dunsford and Boulton (1979), and others, measured the electrical conductivity change in a KHT-seeded wine by the disappearance of free potassium ions (K<sup>+</sup>) from solution. The change in electrical potential (conductivity) was measured with a standard conductivity meter. This "conductivity" test provides a final stable conductivity value that is specific for the wine being tested. Complexing, or "fouling", factors that may be present and possibly affect potassium bitartrate crystal formation and growth are taken into account. The temperature at which the test is performed should be the lowest temperature which the wine is expected to encounter after bottling. During this test, conditions are created for rapid crystal growth if any supersaturation of KHT exists.

An electrical conductivity meter capable of measuring in the range of 100-1000 micro-Siemens is required for this analysis. A Siemens unit is 0.94073 international ohms. Models are also available which are combination conductivity, pH and mV meters.

The Test Procedure. The test procedure outlined by Boulton (1983) is 100 ml of wine chilled to the desired test temperature is mixed and the conductivity measured. One gram of powdered KHT is added and the conductivity is noted at one and two-minute intervals while constantly mixing. The measurement of conductivity is continued until the conductivity reading becomes stable. This, according to Boulton, usually takes less than 20-30 minutes. The conductivity value is affected by temperature; therefore, the sample temperature must be constant throughout the test. If the temperature rises, the conductivity reading will increase simply due to temperature effects.

Stability Criterion. The final conductivity value corresponds to that of the stable wine. It can be used for a comparison with samples taken from the winery during the full-scale stabilization treatment to test when stability has been reached. The difference between the conductivity value before the powdered KHT was added and the final value is a measure of potential KHT instability. If this difference is less than 5% of the initial value, the original wine is considered to be stable. If it is greater than 5% of the initial value, the original wine is unstable and can be treated in the cellar by seeding. Some consider a dry red wine with a conductivity value below 1800 micro mhos/cm or a dry white wine below 1400-1600 to be stable.

#### Cellar Considerations for Contact Seeding

Cooke and Berg (1984) report that approximately ½ of the California wineries they surveyed attempt potassium bitartrate stability by seeding with potassium bitartrate. The most important factors affecting potassium bitartrate stabilization by seeding are:

1) Quantity of KHT and crystal size. The volume of KHT required will depend somewhat on the wine in question, due to the variation in complexing factors. The amount of KHT added for seeding must always constitute an overload to create a supersaturated solution. Table 2 shows the relationship between the quantity of KHT added as seed and several wine components. Tartaric acid, potassium, and the concentration product value (CP) diminish with increased levels of added KHT. This diminution is clear at 4 g/l, then slows with more elevated quantities of KHT (Gumberteau et al. 1981). The range of efficiency has been determined to be between 30 to 150 microns (Gumberteau et al. 1981).

2) Agitation. In the cellar, seeding stabilization should be conducted in a small tank (2000 gallons) where the stabilization temperature and mixing can be controlled. Crystal growth is dependent upon available surface area, and, for this reason, constant agitation is essential. Effective seeding is based upon intense contact of the wine with the powdered KHT added. Table 3 shows the relationship between wine constituents and agitation during the seeding process. The potassium, tartaric acid and CP values are lower in an agitated than a static environment.

3) Time and Temperature. When tartrate seeding is employed, the addition of a large surface area of powdered KHT eliminates the natural energy-consuming nuclei-induction phase and allows for immediate crystal growth. This procedure

Table 2

Influence of quantity of potassium bitartrate (40 micron) added at 0°C and constant agitation.

Conditions of the Assay	Tartaric acid g/l	Potassium mg/l	C.P. x 10 <sup>5</sup>
Control	1.58	920	15.1
+ 1 g/l	1.11	808	9.3
+ 2 g/l	1.03	794	8.5
+ 4 g/l	0.93	765	7.6
+ 8 g/l	0.78	754	6.2

Source: Blouin et al. 1982.

Rhein and Neradt (1979) suggest 4 g/l of 40-micron KHT powder as the optimal quantity and particle size.

Table 3

Influence of agitation on white wine constituents during contact seeding at 0°C with 4 g/l KHT.

Conditions of the Assay	Tartaric Acid g/l	Potassium mg/l	C.P. x 10 <sup>5</sup>
Control	1.58	920	15.1
with agitation	1.17	805	9.8
without agitation	1.38	870	12.5

Source: Blouin et al. 1982

can be carried out at any temperature. When applying the seeding process, the treatment temperature is identical to the desired stability temperature. For example, many wine producers seed their white wines at 0°C and red wines at +5°C. Thus, if the stabilization procedure is correctly performed, wines held at or above these temperatures should remain stable with respect to potassium bitartrate precipitation.

During the first hour of contact seeding there is a rapid reduction in tartaric acid, potassium, and the concentration product value. This reduction slows after the first hour then levels off for most wines at the end of three hours (Blouin et al. 1982). For security, it is desirable to have the duration of contact to be a minimum of 4 hours (Gumberteau et al. 1981). It has been suggested that using 40-micron KHT would allow stabilization in 90 minutes (Neradt 1979). Reduction of the seeding quantity can prolong the stabilization period. Filtration of the wine following contact seeding is essential. This should be performed at the seeding temperature to avoid resolubilization of potassium bitartrate crystals back into solution.

Table 4 shows a comparison of several wine parameters as affected by potassium bitartrate stabilization methods. Gilbert (1976) found the sugar-free extract in KHT-precipitated wines to decrease by 0.8 g/l and total acid to decrease to 0.5 g/l.

Table 4

Sparkling wine cuvee data prior to and after potassium bitartrate stabilization by several methods (Neradt 1979)

Stabilization	ETOH	Sugar-free	Total	Tartaric		
Method	% Vol	Extract	Acid	Acid	K+	
		g/l	pH	g/l	g/l	mg/l
Untreated	9.37	21.26	3.31	7.55	2.50	720
Contact	9.46	20.34	3.20	7.15	1.95	565
Chilling	9.43	21.74	3.23	7.25	2.50	715
Ion Exchange	9.35	20.74	3.26	7.45	2.40	360

Riberaeu-Gayon and Sudraud (1981) report a comparison of potassium bitartrate stabilization techniques using 16 white wines and 11 reds. Each wine was pre-filtered, and potassium bitartrate stabilization was performed by conventional cold stabilization at  $-4^{\circ}\text{C}$  for 14 and 21 days. The results were compared with the contact procedure. Eighty percent of the wines tested showed concentration product values lower for the wines treated by contact seeding. In all tests, contact produced results at least equivalent to conventional cold stabilization.

KHT crystals can be reused after removal from the treated wine. After the crystals have been used repeatedly, they grow in size, decreasing the number of active nuclei and decreasing the crystallization phase (Neradt 1979). Wet grinding of the crystals must be eventually done for optimal performance.

During potassium bitartrate formation, 1.0 g/l of tartaric acid combines with 0.26 gr/l potassium ions (K+) to form 1.26 g/l of potassium bitartrate (KHT). By comparing tartaric acid levels before and after seeding, the wine may be regarded as stable if the differences in tartaric acid at the stabilizing temperature is 200 mg/l or less (SWK 1978). Comparing potassium levels before and after seeding can be used to measure stability. The wine under question may be considered stable if the difference in potassium levels before and after seeding with KHT is 40 mg/l or less (SWK 1978). Comparing the change in titratable acidity before and after seeding can be a gauge for the determination of potassium bitartrate stability. In this case, the wine can be considered stable if the difference in titratable acidity levels before and after seeding is not greater than 100 mg/l.

Ion exchange can also be used to obtain potassium bitartrate stabilization. Ion exchange is generally not considered a premium wine production practice. It is occasionally used when refrigeration fails or in conjunction with refrigeration in order to avoid wine palatability degradation.

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